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(54) Title: DETERGENT COMPOSITIONS			
(57) Abstract			
<p>A particulate detergent composition containing more than 5 wt.% of C₈₋₂₀ primary alcohol sulphate and/or at least 8 wt.% of an ethoxylated C₈₋₂₀ alcohol nonionic surfactant and/or more than 15 wt.% of total non-soap organic surfactant, and a builder system containing alkali metal aluminosilicate, also comprises from 0.01 to 5 wt.% of an antioxidant, preferably a sterically hindered phenol, more preferably butylated hydroxytoluene or butylated hydroxyanisole. The composition exhibits improved stability of the surfactant system on bulk storage and handling.</p>			

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DETERGENT COMPOSITIONSTECHNICAL FIELD

5 The present invention relates to particulate detergent compositions containing high levels of high performance organic surfactants, and especially to compositions containing primary alcohol sulphate or ethoxylated alcohol nonionic
10 surfactants or both. The compositions exhibit improved stability owing to the presence of low levels of antioxidants.

PRIOR ART

15 EP 283 252B and EP 320 219B (Procter & Gamble) disclose particulate detergent compositions in which an antioxidant is incorporated within bleach activator granules, in order to improve bleach activator stability.

20 Various particulate detergent compositions containing antioxidants are also disclosed in JP 50 066 502A/JP 77 003 730B, JP 50 151 905A/JP 78 028 163B and JP 57 128 798A/ JP 84 012 720B (Lion) and in
25 JP 73 008 324B (Daiichi Kogyo Seijaku KK). The benefit is reduced odour and discoloration on storage.

30 It has now been discovered that in particulate detergent compositions containing high surfactant levels and high performance surfactants, especially those containing primary alcohol sulphate and/or ethoxylated nonionic surfactant, improved stability of the surfactant system may be achieved by incorporating antioxidants, especially sterically hindered phenols. The increased safety margin thus achieved
35 facilitates factory handling and bulk storage of these high-active products, thereby providing increased manufacturing flexibility.

DEFINITION OF THE INVENTION

The present invention accordingly provides a particulate detergent composition comprising

- 5 (i) an organic surfactant system comprising anionic and/or nonionic non-soap surfactants, the composition containing more than 5 wt% of C_{8-20} primary alcohol sulphate and/or at least 8 wt% of an ethoxylated C_{8-20} alcohol nonionic surfactant and/or more than 15 wt% of total non-soap organic surfactant,
- 10 (ii) a detergency builder system comprising alkali metal aluminosilicate,
- 15 (iii) from 0.01 to 5 wt% of an antioxidant,
- (iv) optionally other detergent ingredients to 100 wt%,
- 20 all percentages being based on the total composition.

DETAILED DESCRIPTION OF THE INVENTIONThe surfactant system

- 25 The composition of the invention contains a high level or non-soap organic surfactant, preferably 15 wt% or above, preferably from 15 to 25 wt%. However, as indicated below, certain compositions containing total surfactant levels below 15 wt% are also within the scope of the invention.
- 30

- The surfactants may be chosen from anionic, cationic, nonionic, amphoteric and zwitterionic detergent-active compounds, and mixtures thereof. Many suitable detergent-active compounds are available and are fully described in the literature, for example, in "Surface-Active Agents and Detergents", Volumes I and II, by Schwartz, Perry and Berch.
- 35

Anionic surfactants are well-known to those skilled in the art. Examples include alkylbenzene sulphonates, particularly linear alkylbenzene sulphonates having an alkyl chain length of C_8 - C_{15} ; primary and secondary alkylsulphates, particularly C_8 - C_{15} primary alkyl sulphates; alkyl ether sulphates; olefin sulphonates; alkyl xylene sulphonates; dialkyl sulphosuccinates; and fatty acid ester sulphonates. Sodium salts are generally preferred.

Nonionic surfactants that may be used include the primary and secondary alcohol ethoxylates, especially the C_8 - C_{20} aliphatic alcohols ethoxylated with an average of from 1 to 20 moles of ethylene oxide per mole of alcohol, and more especially the C_{10} - C_{15} primary and secondary aliphatic alcohols ethoxylated with an average of from 1 to 10 moles of ethylene oxide per mole of alcohol. Non-ethoxylated nonionic surfactants include alkylpolyglycosides, glycerol monoethers, lactobionamides, and polyhydroxyamides (glucamide).

The invention is especially applicable to compositions containing a surfactant system comprising a C_{8-20} primary alcohol sulphate, an ethoxylated C_{8-20} alcohol nonionic surfactant, or both. Preferred compositions of the invention contain more than 5 wt% of C_{8-20} primary alcohol sulphate, and/or at least 8 wt%, preferably at least 10 wt%, of ethoxylated C_{8-20} alcohol nonionic surfactant. If one or both of these conditions is met, the total level of non-soap organic surfactant in the compositions of the invention need not be greater than 15 wt%, but is however preferably at least 10 wt% and is more preferably at least 12 wt%.

One class of preferred compositions of the invention comprises compositions containing more than 5 wt%, based on the whole product, of C_{8-20} primary alcohol sulphate, optionally in combination with an ethoxylated C_{8-20} alcohol nonionic surfactant.

The preferred compositions may contain at least 8 wt%, more preferably at least 10 wt%, of ethoxylated nonionic surfactant, and advantageously the ratio of primary alcohol sulphate to ethoxylated nonionic sulphate may be less than 1:1. However, compositions containing lesser proportions of nonionic surfactant, for example, those containing additional anionic surfactants, are also within the scope of the invention.

Another class of preferred compositions of the invention, also containing at least 8 wt% and more preferably at least 10 wt% of ethoxylated nonionic surfactant, are those in which the non-soap surfactant system consists wholly or substantially wholly of nonionic surfactants.

The compositions of the invention may also contain fatty acid soap. However, where the limit of at least 15 wt% total surfactant applies, this does not include any fatty acid soap present.

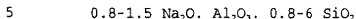
The detergency builder system

The compositions of the invention contain, as detergency builder, an alkali metal aluminosilicate, either alone or in conjunction with one or more supplementary builders.

The total amount of detergency builder in the compositions may suitably range from 5 to 80 wt%, preferably from 10 to 60 wt%.

An essential component of the composition of the invention is an alkali metal, preferably sodium, aluminosilicate builder. Sodium aluminosilicates may generally be incorporated in amounts of from 10 to 70% by weight (anhydrous basis), preferably from 25 to 50 wt%.

The alkali metal aluminosilicate may be either crystalline or amorphous or mixtures thereof, having the general formula:



These materials contain some bound water and are required to have a calcium ion exchange capacity of at least 50 mg CaO/g. The preferred sodium aluminosilicates contain 10 1.5-3.5 SiO_2 units (in the formula above). Both the amorphous and the crystalline materials can be prepared readily by reaction between sodium silicate and sodium aluminate, as amply described in the literature.

15 Suitable crystalline sodium aluminosilicate ion-exchange detergency builders are described, for example, in GB 1 429 143 (Procter & Gamble). The preferred sodium aluminosilicates of this type are the well-known commercially available zeolites A and X, and mixtures thereof.

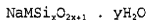
20 The zeolite may be the commercially available zeolite 4A now widely used in laundry detergent powders. However, according to a preferred embodiment of the invention, the zeolite builder incorporated in the compositions of the invention is maximum aluminium zeolite P (zeolite MAP) as 25 described and claimed in EP 384 070A (Unilever). Zeolite MAP is defined as an alkali metal aluminosilicate of the zeolite P type having a silicon to aluminium ratio not exceeding 1.33, preferably within the range of from 0.90 to 1.33, and more 30 preferably within the range of from 0.90 to 1.20.

Especially preferred is zeolite MAP having a silicon to aluminium ratio not exceeding 1.07, more preferably about 1.00. The calcium binding capacity of zeolite MAP is 35 generally at least 150 mg CaO per g of anhydrous material.

The zeolite is advantageously used in conjunction with a supplementary builder. Supplementary organic builders that may be present include polycarboxylate polymers such as polyacrylates, acrylic/maleic copolymers, and acrylic phosphinates; monomeric polycarboxylates such as citrates, gluconates, oxydisuccinates, glycerol mono-, di- and trisuccinates, carboxymethyloxysuccinates, carboxymethyloxymalonates, dipicolinates, hydroxyethyliminodiacetates, alkyl- and alkenylmalonates and succinates; and sulphonated fatty acid salts. This list is not intended to be exhaustive.

Especially preferred supplementary organic builders are citrates, suitably used in amounts of from 5 to 30 wt%, preferably from 10 to 25 wt%; and acrylic polymers, more especially acrylic/maleic copolymers, suitably used in amounts of from 0.5 to 15 wt%, preferably from 1 to 10 wt%.

The compositions of the invention may also contain lamellar crystalline sodium silicate having the composition



wherein M denotes sodium or hydrogen, preferably sodium; x is a number from 1.9 to 4; and y is a number from 0 to 20.

These materials are described in US 4 664 839, US 4 728 443 and US 4 820 439 (Hoechst AG). They are crystalline materials and can easily be characterised by means of their X-ray diffraction patterns. Compared with amorphous silicates, they are significantly better binders of calcium and magnesium ions.

Preferred materials are those in which x = 2, ie compounds of the formula



Both natural and synthetic compounds of this formula are of interest, the synthetic material known as Na-SKS-6 being especially preferred. This material is commercially available from Hoechst AG; its preparation is described, and it is defined in terms of its X-ray diffraction pattern, in the aforementioned US 4 664 839.

The antioxidant

The antioxidant is a material capable of retarding oxidation of the surfactant system of the composition at elevated temperatures, for example, above 100°C.

The antioxidant is preferably present in an amount of from 0.01 to 2 wt%, more preferably from 0.05 to 1 wt% and more especially from 0.1 to 0.5 wt%.

One class of preferred antioxidants is constituted by the sterically hindered phenols.

Suitable antioxidants include the following:

(i) butylated hydroxytoluene (BHT), also known as 2,6-di-tert-butyl p-cresol;

(ii) butylated hydroxyanisole (BHA), also known as (1,1-dimethylethyl)-4-methoxyphenol;

(iii) tert-butylhydroquinone (TBHQ), also known as 1,4-benzenediol.

The optional metal ion sequestrant

Advantageously, the composition of the invention may also contain a sequestrant for transition metal ions.

Preferred sequestrants include aminopolycarboxylates, for example, ethylenediaminetetraacetic acid or its salts; or aminopolyphosphonates, for example, ethylenediamine tetramethylenephosphonic acid (ETDMP) or its salts (Dequest (Trade Mark) 2041 and 2047 ex Monsanto) or higher phosphonates such as diethylenetriamine pentamethylenephosphonic acid (DETPMP) and its salts (Dequest (Trade Mark) 2060 or 2061).

Such phosphonates are conventionally incorporated into detergent compositions containing peroxy bleach compounds, as bleach stabilisers; but in the context of the present invention sequestrants are advantageously present whether or not a bleach system is also present.

The optional bleach system

Compositions of the invention may if desired contain a bleach system. However, the invention is not concerned with bleach stability and is equally applicable to non-bleaching formulations.

Suitable peroxy bleach compounds include organic peroxyacids, and inorganic persalts such as the alkali metal perborates, percarbonates, perphosphates, persilicates and persulphates. Preferred inorganic persalts are sodium perborate monohydrate and tetrahydrate, and sodium percarbonate. The peroxy bleach compound is suitably present in an amount of from 5 to 35 wt%, preferably from 10 to 25 wt%.

The peroxy bleach compound may be used in conjunction with a bleach activator (bleach precursor) to improve bleaching action at low wash temperatures. The bleach precursor is suitably present in an amount of from 1 to 8 wt%, preferably from 2 to 5 wt%. Preferred bleach precursors are peroxycarboxylic acid precursors, more especially peracetic acid precursors and peroxybenzoic acid precursors; and peroxycarbonic acid precursors. An especially preferred bleach precursor suitable for use in the present invention is N,N,N',N'-tetracetyl ethylenediamine (TAED).

As previously indicated, a bleach stabiliser (heavy metal sequestrant) may also be present. Suitable sequestrants are discussed above.

If desired, the bleach system may also include a transition metal bleach catalyst as described and claimed in EP 458 397A, EP 458 398A and EP 509 787A (Unilever).

Other optional ingredients

The compositions of the invention may contain alkali metal, preferably sodium, carbonate, in order to increase detergency and ease processing. Sodium carbonate may suitably be present in amounts ranging from 1 to 60 wt%, preferably from 2 to 40 wt%. However, compositions containing little or no sodium carbonate are also within the scope of the invention.

Powder flow may be improved by the incorporation of a small amount of a powder structurant, for example, a fatty acid (or fatty acid soap), a sugar, an acrylate or acrylate/maleate polymer, or sodium silicate.

One preferred powder structurant is fatty acid soap, suitably present in an amount of from 1 to 5 wt%.

Other materials that may be present in detergent compositions of the invention include sodium silicate; antiredeposition agents such as cellulosic polymers; other soil release or antiredeposition polymers; fluorescers; inorganic salts such as sodium sulphate; lather control agents or lather boosters as appropriate; detergent enzymes, for example, proteases, lipases, cellulases and amylases; dyes; coloured speckles; perfumes; foam controllers; and fabric softening compounds. This list is not intended to be exhaustive.

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Preparation and bulk density

The invention is of especial value for compact detergent powders in which ingredients are in especially close proximity, for example, compositions having bulk densities of 650 g/l or more, and especially of 750 g/l or more.

Detergent compositions of high bulk density are generally prepared either by post-tower densification of spray-dried powder, or by wholly non-spray-drying processes such as mixing and granulation. In both cases a high-speed mixer/granulator may advantageously be used. Processes using high-speed mixer/granulators are disclosed, for example, in EP 340 013A, EP 367 339A, EP 390 251A and EP 420 317A (Unilever).

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Preferred processes for preparing compositions of the invention will include the step of mixing and granulating ingredients in a high-speed mixer/densifier to form a base powder of high bulk density, and optionally postdosing ingredients unsuitable for such processing.

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The antioxidant may either be included in the high bulk density base powder, or postdosed. It is desirable that the antioxidant should be distributed as homogeneously as possible through the final product.

According to one preferred process, a solution or suspension of the antioxidant in nonionic surfactant or in a nonionic surfactant/fatty acid soap mixture is mixed into the base powder before the postdosing of any solid ingredients.

Alternatively, the the antioxidant may be postdosed in particulate form, either as such or in the form of granules also containing a carrier material, for example, zeolite or sodium carbonate, and optionally a binder, for example, sodium silicate or acrylate or acrylate/maleate polymer.

EXAMPLES

The invention is further illustrated by the following non-limiting Examples, in which parts and percentages are by weight unless otherwise stated.

Examples 1 to 6

High bulk density bleaching detergent compositions were prepared to the following general formulation:

	Coconut alcohol sulphate	5.52
	Nonionic 7EO	7.50
10	Nonionic 3EO	5.00
	Zeolite MAP	34.76
	Sodium soap	1.95
	Sodium carbonate	3.13
	Sodium carboxymethylcellulose	0.85
15	BHT	see below
	Percarbonate, TAED etc	30.07
	EDTMP (Dequest 2047)	0.42
	Enzymes	1.42
	Perfume	0.45
20	Foam control/fluorescer granules	3.50
	Water and salts	to 100.00

The composition was prepared as follows. A high bulk density base powder was prepared by mixing and granulating surfactants, zeolite, sodium carbonate and sodium carboxymethylcellulose in a continuous high-speed mixer/densifier (Lödige (Trade Mark) Recycler). Bleach ingredients, further sodium carbonate, enzyme granules and foam control/fluorescer granules were subsequently postdosed. EDTMP was included in the base in some compositions, and postdosed in others.

The BHT was introduced by two different methods:

(a) into the base powder (in the high-speed mixer/granulator)
as a nonionic surfactant/soap/BHT mixture; or

(b) postdosed in powder form.

Compositions were prepared as follows:

<u>Example</u>	<u>Antioxidant</u>	<u>EDTMP</u>
1	0.22% BHT, postdosed	Postdosed
2	0.4% BHT, postdosed	Postdosed
3	0.1% BHT, postdosed	In base
4	0.2% BHT, postdosed	In base
5	0.3% BHT, postdosed	In base
6	0.4% BHT, postdosed	In base

Example 7

A similar formulation was prepared containing sodium perborate instead of sodium percarbonate. It contained postdosed EDTMP, and 0.2% BHT postdosed.

Examples 8 to 10

High bulk density non-bleaching detergent compositions were prepared to the following general formulation:

5	Coconut alcohol sulphate	6.35
	Nonionic 7EO	8.63
	Nonionic 3EO	5.75
	Sodium soap	2.24
10	Zeolite MAP	39.99
	Sodium carbonate	1.26
	Sodium carboxymethylcellulose	0.98
	BHT	see below
	Sodium citrate (2H ₂ O)	22.18
15	EDTMP (Dequest 2047)	1.43
	Enzymes	1.24
	Perfume	0.45
	Foam control/fluorescer granules	3.20
20	Water and salts	to 100.00

The method of preparation was as follows: a high bulk density base powder was prepared as described in Examples 1 to 6; sodium citrate, enzyme granules and foam control/fluorescer granules were subsequently postdosed. EDTMP was postdosed. Compositions were prepared as follows:

<u>Example</u>	<u>Antioxidant</u>		<u>EDTMP</u>
30	8	0.30% BHT	postdosed
	9	0.1% BHT + 0.1% BHA	postdosed
	10	0.05% BHT + 0.05% BHA	postdosed

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CLAIMS

1 A particulate detergent composition comprising an organic
5 surfactant system comprising anionic and/or nonionic non-soap
surfactants, a detergency builder system and other detergent
ingredients to 100 wt%, characterised in that:

10 (i) the composition contains more than 5 wt% of C_{8-20}
primary alcohol sulphate and/or at least 8 wt% of an
ethoxylated C_{8-20} alcohol nonionic surfactant and/or more
than 15 wt% of total non-soap organic surfactant,

15 (ii) the detergency builder system comprises alkali metal
aluminosilicate,

(iii) the composition further comprises from 0.01 to
5 wt% of an antioxidant,

20 all percentages being based on the total composition.

2 A detergent composition as claimed in claim 1,
25 characterised in that the organic surfactant system comprises
 C_{8-20} primary alcohol sulphate and ethoxylated C_{8-20} alcohol
nonionic surfactant.

30 3 A detergent composition as claimed in claim 2,
characterised in that the composition contains more than 5 wt%
of primary alcohol sulphate.

35

- 4 A detergent composition as claimed in claim 2,
characterised in that the composition contains more than
15 wt% of the surfactant system (i), which system comprises
C₈₋₂₀ primary alcohol sulphate and ethoxylated C₈₋₂₀ alcohol
5 nonionic surfactant.
- 5 A detergent composition as claimed in claim 2,
10 characterised in that the surfactant system (i) comprises C₈₋₂₀
primary alcohol sulphate and ethoxylated C₈₋₂₀ alcohol
nonionic surfactant in a ratio of less than 1:1.
- 15 6 A detergent composition as claimed in claim 1,
characterised in that the composition comprises at least
10 wt% of ethoxylated nonionic surfactant.
- 20 7 A detergent composition as claimed in claim 1,
characterised in that the antioxidant (iii) is a sterically
hindered phenol.
- 25 8 A detergent composition as claimed in claim 7,
characterised in that the antioxidant (iii) is selected from
30 butylated hydroxytoluene, butylated hydroxylanisole, and tert-
butylhydroquinone.
- 35

9 A detergent composition as claimed in claim 1,
characterised in that it comprises from 0.05 to 2 wt% of
antioxidant (iii).

5

10 A detergent composition as claimed in claim 1,
characterised in that it also comprises a sequestrant for
transition metal ions.

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11 A detergent composition as claimed in claim 10,
characterised in that the sequestrant comprises an
aminopolycarboxylate or aminopolyphosphonate.

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12 A detergent composition as claimed in claim 10,
characterised in that the sequestrant comprises
ethylenediamine tetramethylenephosphonic acid or a salt
thereof.

20

25

13 A detergent composition as claimed in claim 1,
characterised by a bulk density of at least 650 g/l.

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14 A detergent composition as claimed in claim 1,
characterised in that it is not the product of a spray-drying
process.

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- 15 A process for the preparation of a particulate detergent composition as claimed in claim 1, which includes the step of mixing and granulating ingredients in a high-speed mixer/densifier to form a base powder of high bulk density and optionally postdosing ingredients unsuitable for processing in a high-speed mixer/densifier, characterised in that a solution or suspension of the antioxidant in nonionic surfactant is mixed with the base powder before the postdosing of any solid ingredients.
- 10
- 16 A process for the preparation of a particulate detergent composition as claimed in claim 1, which includes the step of mixing and granulating ingredients in a high-speed mixer/densifier to form a base powder of high bulk density and optionally postdosing ingredients unsuitable for processing in a high-speed mixer/densifier, characterised in that the antioxidant is postdosed in particulate form.
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INTERNATIONAL SEARCH REPORT

International Application No.
PCT/EP 95/02709

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C11D3/00 C11D3/20 C11D17/06

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP,A,0 593 014 (KAO CORP) 20 April 1994 see page 8, line 50 - line 54; claim 1 ---	1,6-9
A	EP,A,0 320 219 (PROCTER & GAMBLE) 14 June 1989 cited in the application see claims 1-4,21; examples ---	1,7-12
A	EP,A,0 283 252 (PROCTER & GAMBLE) 21 September 1988 cited in the application see claims 1-3,20; examples ---	1,7-12
A	US,A,4 088 594 (FERNLEY GEORGE W ET AL) 9 May 1978 see column 1, line 36 - column 2, line 40; claims --- -/--	1,7-9

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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Date of the actual completion of the international search

12 January 1996

Date of mailing of the international search report

19.01.96

Name and mailing address of the ISA

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INTERNATIONAL SEARCH REPORT

Patent Application No.
PCT/EP 95/02709

C (Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	NL,A,7 411 994 (UNILEVER NV) 19 March 1975 see claims; examples 1,2 -----	1,7-9

INTERNATIONAL SEARCH REPORT

Information on patent family members

Int: International Application No
PCT/EP 95/02709

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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		DE-T- 3888116	04-08-94
		ES-T- 2061692	16-12-94
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		US-A- 4853143	01-08-89
		DE-D- 3888116	07-04-94
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		NL-A- 7613955	21-06-77
		SE-B- 425000	23-08-82
		SE-A- 7614248	20-06-77
NL-A-7411994	19-03-75	NONE	

